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Datasheet for the decision of 11 January 2011

T 2371/09 - 3.2.07 Case Number:

Application Number: 00304154.8

Publication Number: 1055741

IPC: C23C 10/00

Language of the proceedings: EN

Title of invention:

Fabrication of superalloy articles having hafnium- or zirconium-enriched protective layer

Applicant:

GENERAL ELECTRIC COMPANY

Headword:

Relevant legal provisions:

EPC Art. 56, 113(1) EPC R. 115(2)

Relevant legal provisions (EPC 1973):

Keyword:

- "Decision according to the state of the file"
- "Inventive step (main and auxiliary request no)"

Decisions cited:

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 2371/09 - 3.2.07

DECISION
of the Technical Board of Appeal 3.2.07
of 11 January 2011

Appellant: GENERAL ELECTRIC COMPANY

1 River Road Schenectady NY 12345 (US)

Representative: Szary, Anne Catherine

London Patent Operation GE International Inc. 15 John Adam Street London WC2N 6LU (GB)

Decision under appeal: Decision of the Examining Division of the

European Patent Office posted 6 July 2009

refusing European patent application

No. 00304154.8 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: H. Meinders Members: H. Hahn

I. Beckedorf

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Summary of Facts and Submissions

- I. The applicant lodged an appeal against the decision of the Examining Division to refuse the European patent application No. 00 304 154.8.
- II. In the present decision the following documents of the examination proceedings are cited:

D1 = WO-A-95 30779

D2 = US-A-4 880 614

- III. The Examining Division held that the subject-matter of claim 1 of the single request filed with letter of 16 March 2009 lacked an inventive step with respect to a combination of the teachings of the closest prior art D1 with D2.
- IV. With its grounds of appeal the appellant requested to set aside the decision and to grant a patent either on the basis of the claims 1-9 of the main request as underlying the impugned decision or the claims 1-9 of the auxiliary request as filed together with the grounds of appeal. As an auxiliary request oral proceedings were requested.
- V. With a communication dated 21 September 2010 and annexed to the summons to oral proceedings the Board gave its preliminary and non-binding opinion with respect to the claims of those requests.

The Board stated amongst others that the term "about" used in connection with the hafnium and zirconium content rendered claims 1, 5 and 6 of both requests

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unclear which therefore appeared to contravene
Article 84 EPC. Furthermore, claim 1 of both requests
appeared not to be supported by the description as
required by Article 84 EPC.

With respect to the issue of inventive step the Board stated:

"4. Inventive step (Article 56 EPC)

Main request

D1 discloses a method for improving oxidation and spallation resistance of diffusion aluminide coatings by adding from 0.01 to 0.30 weight percent of an additive selected from the group consisting of zirconium, yttrium and mixtures thereof to nickel-base super alloys which then bear a diffusion aluminide coating (see abstract; and page 3, lines 3 to 21; page 8, line 27 to page 9, line 10). The aluminide coating is directly applied to the substrate by conventional techniques (see page 7, lines 1 to 19; and claim 1). According to D1 this teaching is applicable to both single crystal materials and polycrystalline substrates (see page 4, lines 24 and 25). The zirconium content is preferably 0.02-0.15 weight percent (see claim 2). D1 further mentions that said aluminide layer is provided on the surface of the substrate, in the absence of any further coating (see page 3, lines 7 to 9 and claims 1 and 14). D1 additionally remarks that "if hafnium is present in the alloy of the substrate, the effectiveness of the yttrium is enhanced" (see page 3, lines 21 and 22). According to the examples a diffusion heat treatment of the aluminide coated

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superalloy articles at 1975±25°F (= 1079.4± 3.9°C) was carried out (see page 8, lines 18 to 20) which implies that said zirconium will have been interdiffused from the substrate into the aluminide layer.

The appellant's arguments - that D1 teaches away from further coating layers - do not appear to hold for the following reasons:

It is true that D1 teaches to directly apply the aluminide layer to the nickel-base superalloy substrate with no intermediate coatings and with no further coating thereupon (see page 3, lines 7 to 9 and claims 1 and 14). It needs, however, to be considered that an aluminium oxide layer will form on said aluminide layer at least during the use in the operating environment of such an aluminide coated superalloy gas turbine part. Furthermore, although D1 explicitly does not suggest an additional TBC layer it appears to be reasonable to assume that the person skilled in the art, being familiar with aluminide coatings and knowing that they are commonly used to improve the adhesion of a subsequently applied TBC layer (this knowledge belongs to the common general knowledge of the person skilled in the art; see e.g. D2), would - in order to increase the operating temperature and to improve the life time of the (implied) nickel-base superalloy components for high temperature gas turbine engines according to D1 having the aluminide coating directly applied (see page 1, lines 8 to 16) - at least try to deposit an additional TBC layer on the mentioned single crystal superalloy substrates - which have a higher operating temperature than the polycrystalline ones (compare e.g. D2,

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column 1, lines 42 to 47) - in order to reduce the temperature of the TBC coated single crystal nickel-base superalloy substrate. There exists no prejudice to do so nor can the mention in D1, that no such further coating is applied be considered as such a prejudice. To the contrary, it belongs to the general knowledge that the aluminide coating when oxidized produces an alumina layer which represents a very good bond coat for a subsequently deposited ceramic TBC layer, such as the commonly used yttria stabilised zirconia coating. Thereby the person skilled in the art would, however, arrive at the subject-matter of claim 1 of the main request without any inventive skill.

Auxiliary request

On the one hand it is true that D1 does not mention a hafnium content of from about 0.2 to about 2.0 weight percent Hf, on the other hand D1 remarks that "if hafnium is present in the alloy of the substrate, the effectiveness of the yttrium is enhanced" (see page 3, lines 21 and 22). Thus there is the clear teaching in D1 to add hafnium to superalloy compositions comprising yttrium in order to increase the effectiveness of the yttrium which implies that the person skilled in the art would only have to make some experiments in order to determine the optimum concentration range for Hf in order to obtain this enhancement of the yttrium effectiveness. Thereby the person skilled in the art would, however, arrive at the subject-matter of claim 1 of the auxiliary request without any inventive skill."

VI. With letter dated 23 December 2010 the appellant submitted, as a response to the summons to oral

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proceedings, two amended sets of claims as a new main request and new first auxiliary request - which claims differ from those of the former requests only in that the objected term "about" has been deleted from claims 1, 5 and 6 - in combination with an amended description for each request being supported by explanations of the amendments carried out which were made to overcome the clarity and support objections raised by the Board. Finally the appellant stated therein "It is requested that the appeal proceed on the basis of the new sets of claims and new pages filed herewith".

This letter did **not** contain any argument concerning inventive step of the subject-matter claimed.

- VII. With letter dated 10 January 2011 the appellant informed the Board that it would not be represented at the oral proceedings and further requested a decision based on the prior written proceedings, i.e. including the requests submitted with letter dated 23 December 2010.
- VIII. At the end of the oral proceedings held on 11 January 2011 in the absence of the appellant, the Board announced its decision.
- IX. Independent claim 1 of the new main request reads as follows (deletions compared to the main request dated 16 March 2009 are marked in strikethrough; emphasis added by the Board):
 - "1. A method for preparing a single-crystal superalloy article having a protective layer (34) thereon,

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comprising the steps of

selecting a nominal nickel-base superalloy
composition;

preparing a modified nominal nickel-base superalloy composition, wherein the modified nominal nickel-base superalloy composition has an excess of a protective-layer modifying element over that of the nominal nickel-base superalloy composition, the protective-layer modifying element being selected from the group consisting of

from $\frac{\text{about}}{\text{about}}$ 0.2 to $\frac{\text{about}}{\text{about}}$ 2.0 percent by weight hafnium, and

from about 0.1 to about 0.5 percent by weight zirconium, and combinations thereof;

processing the modified nominal nickel-base superalloy composition into a substrate (32) having the shape of the article and being substantially a single crystal;

applying a protective layer (34) to a surface (36) of the substrate (32), the as-applied protective layer (34) having the protective-layer modifying element in a lower concentration than the substrate (32);

depositing a ceramic layer overlying the protective layer (34); and

interdiffusing the protective-layer modifying element from the substrate (32) into the applied protective layer (34)."

X. Independent claim 1 of the new auxiliary request reads as follows (deletions compared to the former auxiliary request are marked in strikethrough; emphasis added by the Board): - 7 - T 2371/09

"1. A method for preparing a single-crystal superalloy article having a protective layer (34) thereon, comprising the steps of

selecting a nominal nickel-base superalloy
composition;

preparing a modified nominal nickel-base superalloy composition, wherein the modified nominal nickel-base superalloy composition has an excess of a protective-layer modifying element over that of the nominal nickel-base superalloy composition, the protective-layer modifying element being selected from the group consisting of from about 0.2 to about 2.0 percent by weight hafnium, and

combinations thereof with from about 0.1 to about 0.5 percent by weight zirconium;

processing the modified nominal nickel-base superalloy composition into a substrate (32) having the shape of the article and being substantially a single crystal;

applying a protective layer (34) to a surface (36) of the substrate (32), the as-applied protective layer (34) having a lower concentration of the protective-layer modifying element than the substrate (32); and

interdiffusing the protective-layer modifying element from the substrate (32) into the applied protective layer (34)."

Reasons for the Decision

1. On considering the case at the oral proceedings, duly held pursuant to Rule 115(2) EPC in the absence of the appellant, the Board came to the conclusion that the subject-matter of the claims 1 of the new main and new

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auxiliary request - which differ from the former two requests only in that the objected terms "about" have been deleted therefrom (see points IX and X above) - lacked an inventive step for the reasons already set out in the Board's communication.

Since the Board came to the conclusion that the subject-matter of claims 1 of the two requests lacks an inventive step, there was no need to verify whether or not these claims comply with Articles 84 and/or 123(2) EPC.

- 2. Furthermore, the Board interpreted the appellant's statement in its letter dated 10 January 2011 that it did not intend to attend the oral proceedings and its request to decide on the state of the file as a withdrawal of the auxiliary request for oral proceedings, as is consistent Case Law (see Case Law of the Boards of Appeal, 6th edition 2010, VI.C.2.2), i.e. the appellant relies on its written submissions only.
- In the communication accompanying the summons for oral proceedings the Board, taking account of these substantive submissions, amongst others raised objections under Article 56 EPC, explaining why in the Board's opinion the subject-matter of claim 1 of the main request and of claim 1 of the auxiliary request lacked an inventive step over the teaching of D1 in combination with the common general knowledge of the person skilled in the art as e.g. represented by D2 (see point V above).
- 4. The appellant did not reply in substance to these objections which continue to fully apply to the

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subject-matter of claim 1 of the new main and auxiliary request (see point VI above). Since there has been no attempt by the appellant to refute or overcome the lack of inventive step objections raised in the above communication, the Board has no reason to depart from its preliminary opinion expressed therein.

- 4.1 The Board further remarks that the definitions of claim 1 of the new main request "the protective-layer modifying element being selected from the group consisting of from 0.2 to 2.0 percent by weight hafnium, and from 0.1 to 0.5 percent by weight zirconium, and combinations thereof" and of claim 1 of the new auxiliary request "the protective-layer modifying element being selected from the group consisting of from 0.2 to 2.0 percent by weight hafnium, and combinations thereof with from 0.1 to 0.5 percent by weight zirconium" do not exclude that the element yttrium - which according to D1 represents a modifying additive element for the nickel base superalloy substrate and which then diffuses from the superalloy substrate through the applied aluminide coating to the surface thereof (see page 4, lines 5 to 16 and page 8, line 27 to page 9, line 10) - can be present in the nominal nickel base superalloy composition according to the present application (compare in this context the preferred nominal nickel base superalloys which specify yttrium contents as defined in claims 5 and 6 of the main and auxiliary request).
- In this context the Board remarks that it was intended to discuss at the oral proceedings the above mentioned additional argument with respect to yttrium. Due to the appellant's decision not to appear, it has waived the

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opportunity to be heard on this issue (Article 113(1) EPC), see also the Case Law of the Boards of Appeal, $6^{\rm th}$ edition 2010, VI.B.3 and VI.B.3.2.

5. With regard to the above the Board concludes - for the reasons set out in point 4 of the communication (see point V above) - that the subject-matter of claim 1 of the main request and of claim 1 of the auxiliary request lack inventive step over D1 and the common general knowledge of the person skilled in the art. The two requests are therefore not allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

G. Nachtigall H. Meinders